

One-Ångstrom Microscope Report:

Defocus step size of the LBNL One Ångstrom Microscope

Michael A. O’Keefe* and E. Chris Nelson**

*Materials Sciences Division, LBNL B2-200, One Cyclotron Road, Berkeley, CA 94720, USA

**NCEM, Materials Sciences Division, LBNL B72, One Cyclotron Road, Berkeley, CA 94720

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One-Ångstrom Microscope Report:**Defocus step size of the LBNL One Ångstrom Microscope****Michael A. O'Keefe and E. Chris Nelson****Abstract**

Change in focus of a high-resolution electron microscope is generally assumed to be linear with change in objective lens current. Thus the defocus step size should be constant for a constant step in lens current. Measurements on the LBNL One-Ångstrom Microscope show that the step size increases with increasing underfocus (reduced lens current). Differentiation of a best-fit quadratic shows that the defocus step size varies linearly as defocus changes.

Introduction

The resolution of the high-resolution transmission electron microscope is limited by its spherical aberration coefficient. In the case of the Philips CM300FEG/UT this limit is about 1.7Å. The aim of the NCEM One-Ångstrom Microscope project is to achieve sub-Ångstrom resolution by extending the resolution of a modified high-resolution microscope to its information limit [1].

The information limit of the standard CM300FEG/UT is 1.05Å [2], whereas that of the OÅM (a modified CM300FEG/UT) is below 0.8Å [3]. In order to attain a resolution on the OÅM out to its information limit, we run the Philips/Brite-Euram software for focal-series reconstruction by Coene and Thust [4,5]. This software accepts a focal-series of high-resolution images and generates an estimate of the (complex) exit-surface wave – this estimate will be limited to the resolution of the information limit of the focal series.

The software requires the defocus values of the images in the focal series, input as a starting defocus and a defocus step size. Gatan software was used to measure the defocus of each image from its diffractogram. However, the presence of Bragg spots in the diffractogram made it difficult to obtain accurate values of defocus with this software.

Since it is easier to determine the defocus from a diffractogram without strong spots, a series of images of an amorphous carbon specimen was obtained and used to calibrate the defocus of the microscope.

References

- [1] M.A. O'Keefe, *LBNL Symposium on Microstructures of Materials*, ed. K. Krishnan (1993) 121-126.
- [2] Hans Bakker, Arno Bleeker, and Peter Mul, *Ultramicroscopy* **64** (1996) 17-34.
- [3] M.A. O'Keefe, E.C. Nelson, Y.C. Wang and A. Thust, *Philosophical Magazine B* **81** (2001) 11: 1861-1878.
- [4] W.M.J. Coene, A. Thust, M. Op de Beeck and D. Van Dyck, *Ultramicroscopy* **64** (1996) 109-135.
- [5] A. Thust, W.M.J. Coene, M. Op de Beeck and D. Van Dyck, *Ultramicroscopy* **64** (1996) 211-230.

Experimental Procedure

On the microscope, the nominal focus step is 506pm for focus step 1 and 2.02nm for focus step 2. With the carbon specimen inserted in the OAM, defocus was adjusted and the diffractogram examined on-line until the best possible Scherzer-defocus image was obtained. Defocus was then taken towards overfocus by +330Å (nominal) and the indicated microscope defocus value was set to zero.

From the zero (nominal) value, defocus was taken underfocus to the extended Scherzer value of -420Å. This setting was confirmed by the observation of a “split” main passband in the diffractogram. The objective lens current at this setting was 12578mA.

Since the defocus range for focal-series reconstruction generally does not usually extend beyond -4000Å underfocus, we next set the microscope defocus to an estimated value of -4000Å. At this setting we measured the actual defocus as -3922Å.

We set the indicated defocus on the microscope to -3922Å and stepped the defocus back in nominal steps of 202Å (10 steps of 2.02nm) using focus step 2 and measuring the actual defocus at each 202Å (nominal) step from the diffractogram.

Experimental Results

The measured defocus value changed more rapidly than the nominal value given by the microscope. After 13 steps the indicated defocus was -1296Å, but the measured defocus was -788Å (table).

<u>Step</u>	<u>Nominal</u>	<u>Actual</u>
0	-3922	-3922
10	-3720	-3697
20	-3518	-3409
30	-3316	-3140
40	-3114	-2874
50	-2912	-2678
60	-2710	-2413
70	-2508	-2138
80	-2306	-1928
90	-2104	-1709
100	-1902	-1499
110	-1700	-1259
120	-1498	-1033
130	-1296	-788

Instead of changing by 2626Å, as expected for a defocus step of 2.02Å per “click” of focus step 2, the actual defocus changed by 3134Å, indicating a calibration factor of about 1.19x.

The measured values of defocus were plotted as a function of the nominal defocus and a straight line was fitted in order to determine a more accurate calibration factor for the microscope step size (figure 1).

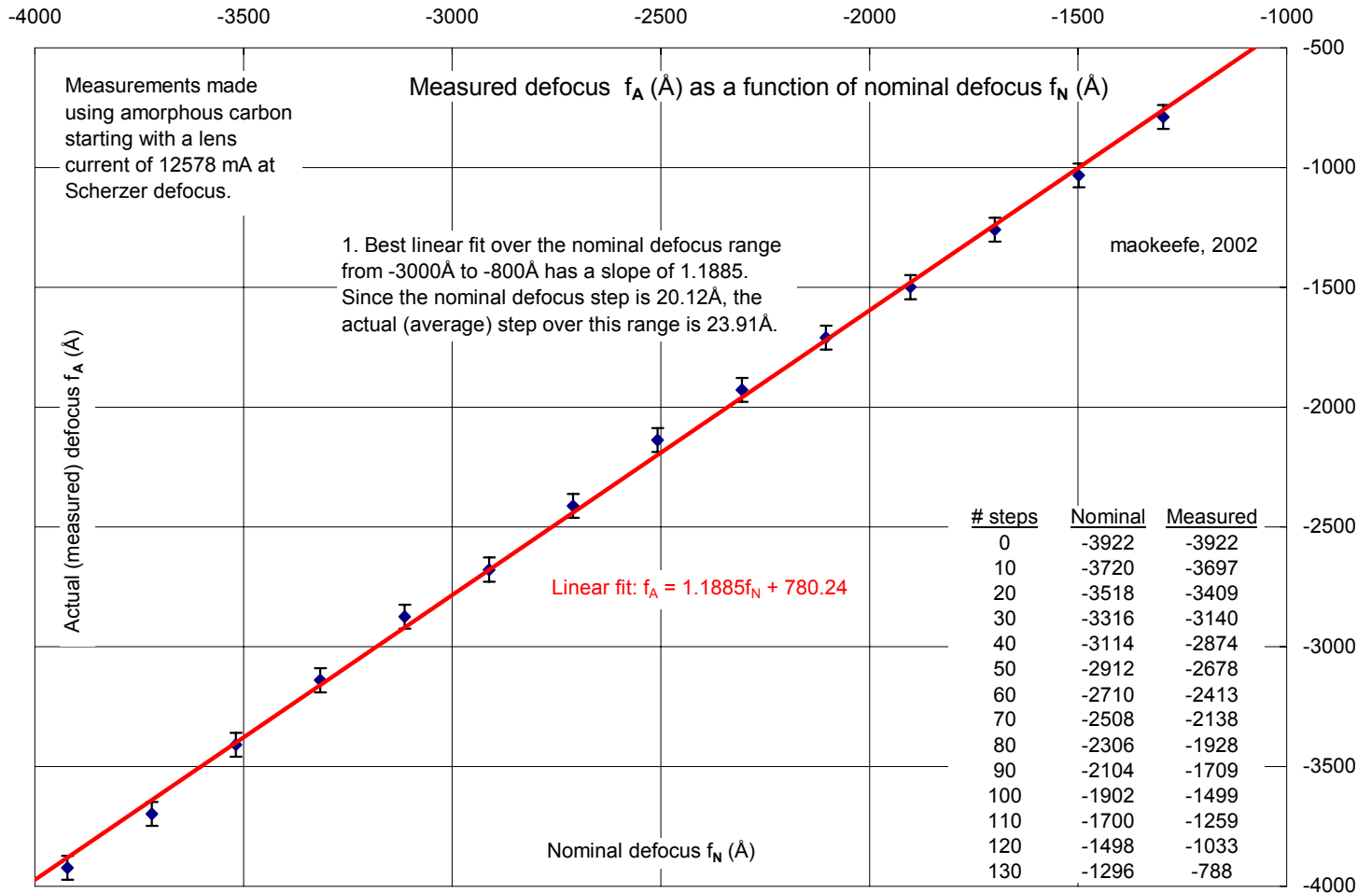


Figure 1 – plot of measured defocus values (over the range from -4000 Å to -500 Å) as a function of indicated defocus (over the range from -4000 Å to -1000 Å) for the NCEM OAM. Straight-line fit has slope of 1.19 indicating that the actual defocus step is 19% higher than the nominal value.

The slope of the best-fit line is 1.188, so the average measured defocus step (for 10 clicks of focus step 2) is 23.77 Å instead of the nominal 20.2 Å.

Interestingly, the mid-range points in the figure are above the line of best fit, while those at both end points are below it.

Since there appeared to be a parabolic component to the best-fit curve, a fit was made for a general quadratic (figure 2). Then the best-fit slope varies from 1.324 to 1.063 over the range from -3900\AA to -1300\AA (nominal, corresponding to -3900\AA to -890\AA actual).

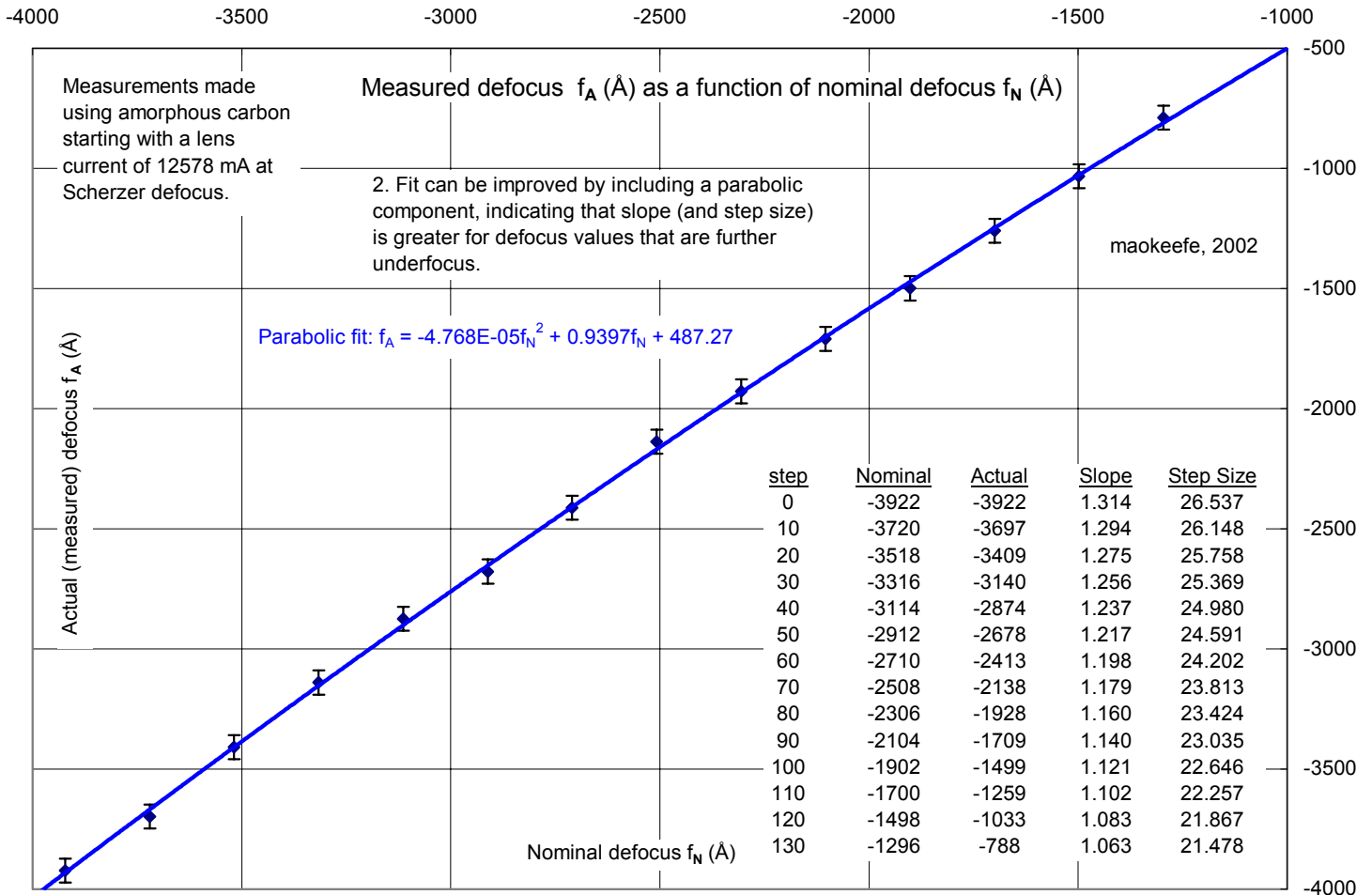


Figure 2 – plot of measured defocus values (over the range from -4000\AA to -500\AA) as a function of indicated defocus (over the range from -4000\AA to -1000\AA) for the NCEM OÅM. Quadratics fit has slope from 1.314 to 1.063 corresponding to step sizes ranging from 26.54\AA to 21.48\AA .

Differentiation of the best-fit quadratic shows that the step size varies linearly from 26.54\AA to 21.48\AA (figure 3).

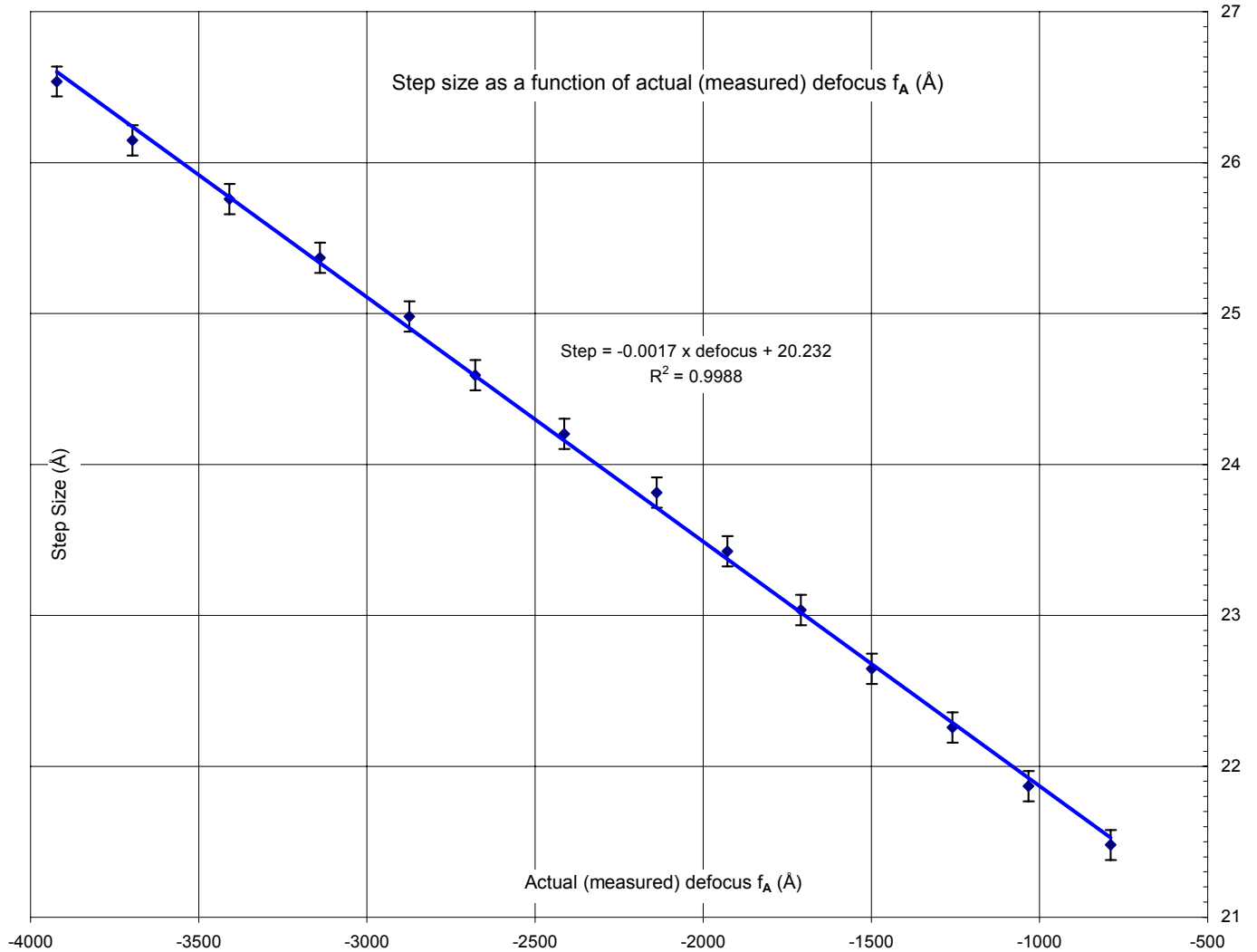


Figure 3 – plot of step size as a function of measured defocus (from -4000\AA to -1000\AA) for the NCEM OAM. Quadratics fit gives slopes from 1.314 to 1.063 corresponding to step sizes ranging from 26.54\AA to 21.48\AA .

Summary: The change in focus of a high-resolution electron microscope is generally assumed to be linear with change in objective lens current. Thus the defocus step size should be constant for a constant step in lens current. Measurements on the LBNL One-Angstrom Microscope show that the step size increases with increasing underfocus (reduced lens current). Differentiation of the best-fit quadratic shows that the defocus step size varies linearly from 26.54\AA to 21.48\AA . over a defocus range from -3900\AA to -800\AA .